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**ENVIRONMENTAL QUALITY ASSESSMENT OF THE
IROQUOIS NATIONAL WILDLIFE REFUGE
ALABAMA, NEW YORK
1988 - 1990**

89-5-104



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Executive Summary

The U.S. Fish and Wildlife Service conducted a 3-year monitoring program at the Iroquois National Wildlife Refuge, Alabama, New York to identify any contaminant problems that could potentially effect the health and well being of a breeding pair of bald eagles (*Haliaeetus leucocephalus*), an endangered species, that have produced a pair of fledglings annually for the past 4 years.

Samples of sediments, snapping turtle (*Chelydra serpentina*) tissues, wood duck (*Aix sponsa*) and hooded merganser (*Lophodytes cucullatus*) eggs, and fish were collected at various locations on the refuge and two contiguous New York State Wildlife Management Areas. All samples were analyzed for chlorinated hydrocarbon compounds and heavy metals/trace elements.

All hydrocarbon compounds tested for were either not detected or were measured at levels that do not pose an apparent threat to the eagles. The levels of inorganic elements in sediments exhibited considerable variation, as expected, but no serious elevations were apparent. Inorganic levels in the various tissue matrices were virtually all below levels of concern.

Based on the results obtained, it is concluded that no significant threat to the eagles from environmental contaminants exists at this time. A recommendation has been made that samples of fish, the primary food of the eagles, be periodically collected and analyzed for contaminants of concern.

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Acknowledgements

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The Pennsylvania Field Office, State College, PA, graciously provided the electrofishing boat for the fish collections and the assistance of staff members Teresa Mackey and Adam Smith was greatly appreciated.

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ENVIRONMENTAL QUALITY ASSESSMENT AT THE
IROQUOIS NATIONAL WILDLIFE REFUGE
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1988 - 1990

INTRODUCTION

In 1988 a contaminant monitoring and surveillance program was initiated at Iroquois National Wildlife Refuge (Iroquois NWR), Alabama, New York. The refuge is the home of a breeding pair of endangered bald eagles who have successfully reared a pair of eaglets in each of the past 4 years. The contaminant survey was begun for the purpose of assessing the eagles' exposure to environmental contaminants and to establish an environmental contaminant profile for the refuge complex to be used as a data baseline for comparison with future monitoring efforts.

Initially, only the snapping turtle was screened for contaminants. Pooled samples of liver, fat, and egg tissue were collected from turtles captured on the refuge. These tissues were analyzed for organochlorine compounds, polynuclear aromatic hydrocarbons (PAH), and heavy metals. The results were summarized in a preliminary report (McCartney, 1990).

The initial data showed low levels of organic and inorganic contaminants suggesting that the refuge was relatively clean with respect to many contaminants of concern. There was, however, just enough mercury and selenium measured in egg and/or liver tissue to warrant further investigation inasmuch as pooling tissues results only in an average value with no clue to the range and magnitude of individual values for a given contaminant.

In 1989 the survey area was expanded to include the State of New York's Oak Orchard and Tonawanda Wildlife Management Areas, which adjoin the refuge on the east and west respectively, and included sediment samples, eggs of the wood duck and hooded merganser and fat and liver tissue from snapping turtles. Tissue samples from individual turtles were submitted in lieu of pooling.

The analytical results for the 1989 samples were not received prior to initiation of the 1990 sampling schedule so this report incorporates the results of all sampling to date and completes the three-year baseline evaluation of the contaminant profile at the Iroquois National Wildlife Refuge.

Sampling in 1990 included additional sediment samples from tributaries to the entire complex and fish collections from the refuge only. In addition, the Service has simultaneously been pursuing the recovery of a small quantity of the herbicide 2,4,5-trichlorophenoxy acetic acid (2,4,5-T) buried more than 20 years ago at the refuge. Since the manufacture of 2,4,5-T often resulted in contamination of the parent material with traces of the dioxin family of compounds, separate sediment samples for analysis of the dioxin isomer

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2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) were obtained from selected sites on the refuge. 2,3,7,8-TCDD is regarded as the most toxic of the dioxin compounds. These results are also reported here.

The rationale for analyzing sediments is obvious since they represent a potential reservoir for any contaminants entering the refuge complex from the surrounding watersheds over an extended period of time.

The choice of wood duck eggs was based on published information that their egg chemistry may be influenced by nutrients consumed by the female on the breeding ground just prior to laying. Drobney (1982) found that wood duck hens in southeastern Missouri put on nearly all their weight gain on the breeding grounds just prior to laying and that the most intensive period of fat deposition by hens also occurs just before laying. He found that by the time the clutch was completed the females had depleted virtually all of their visceral fat and almost three-fourths of their carcass fat. Thus, the contaminant profile of the wood duck egg should reflect the quality of the forage base at the refuge complex and not what the hens may have accumulated elsewhere between breeding seasons.

Hooded merganser eggs were included for two reasons. The hens often use wood duck boxes for nesting so their eggs are easily obtainable simultaneously with the wood duck eggs, and there is a considerable amount of historic data for comparison of eggshell thickness and contaminant content, including some from the Iroquois Refuge.

Wood ducks feed primarily on vegetative material but the laying hen increases the percentage of invertebrates in her diet from 45% to 82% during the interval between the pre-breeding period and the peak of the laying period (Drobney, 1984). On the other hand the hooded merganser is primarily a fish and invertebrate eater. Thus, the egg chemistry of the respective species could reflect the quality of two different trophic levels, albeit with some overlap, at the combined Iroquois Refuge and New York State Wildlife Management complex.

The fish samples represent the food source that the bald eagles directly utilize and their contaminant profile would most reflect the potential for eagle exposure to contaminants.

METHODS

Snapping Turtles

Between April 26 and June 20, 1989, refuge and New York State Department of Environmental Conservation (DEC) personnel collected a total of 15 snapping turtles from the combined Federal and State areas. Five turtles were collected within each of the 3 areas, Iroquois NWR (IR), Oak Orchard Wildlife Management Area (OOWMA), and Tonawanda Wildlife Management Area (TOWMA). The turtles were tagged with the date and location of capture and stored in deep freeze at the refuge. At the end of June, the turtles were autopsied and individual samples of fat and liver were submitted for analysis.

Sediments

Sediment samples from the management pools were collected on July 28, 1989. Two samples were collected at the OOWMA (at Goose Pond and North Marsh), two at the Iroquois NWR (Ringneck Marsh and Oneida Marsh), and two at the TOWMA (Cinnamon Marsh and Wood Marsh). Tributary sediment samples were collected on June 6, 1990 at six locations around the periphery of the complex. See the map set (Figures 1, 1a, and 1b) for locations of all sediment, egg, and fish collections.

Sediments for 2,3,7,8-TCDD analysis were collected at 4 sites on the Iroquois Refuge on August 30, 1990. Samples were obtained from the Onondaga Pool (1), the Mohawk Pool (2), and the Oneida Pool (1).

Samples were collected, handled, and stored using standard methods and protocols. See Appendix A.

Waterfowl Eggs

Wood duck eggs and hooded merganser eggs were collected and processed during the week of April 10-14, 1989. Three clusters of nest boxes were selected on each of the three management areas (See Figures and Appendix B). At each cluster site, boxes were checked randomly until wood duck eggs were found. One egg per box was taken until 3 eggs were collected at each site. A total of 27 wood duck eggs were collected (9 total sites, 3 eggs per site). If hooded merganser eggs were encountered, either in the same box with wood duck eggs or alone in separate boxes at the same cluster site, one per box was collected until 3 were collected. Since merganser eggs occurred much less frequently in the boxes, some sites did not provide a full complement of their eggs. No attempt was made to collect merganser eggs from areas other than the pre-selected sites. A total of 14 hooded merganser eggs were collected (4 sites produced 3 eggs each and 1 site produced only 2 eggs).

We attempted to collect the wood duck eggs as early in the egg laying process as possible as Drobney (1982) estimated that visceral fat deposits were depleted by the time the fifth egg was laid. However, in a few instances it was necessary to take an egg where a clutch could be found, regardless of the clutch size, due to the scarcity of nesting pairs. Consequently, it should be recognized that egg composition data does not take into account possible variability within, or among, nests. Nor does it take into account that hooded merganser hens sometimes distribute eggs among several different nests (Zicus et al., 1988), thus the 14 merganser eggs may not necessarily represent 14 different hens.

All eggs were refrigerated and processed within 24 hours of collection. Measurements of whole egg mass, width, length, and volume were recorded for each egg. Length and width measurements were taken with a caliper, egg volume was obtained using a water displacement technique, and whole egg mass was recorded to the nearest 100th of a gram using an OHAUS "Port-O-Gram" electronic digital read-out balance.

Figure 1. Sampling Locations at Iroquois National Wildlife Refuge.

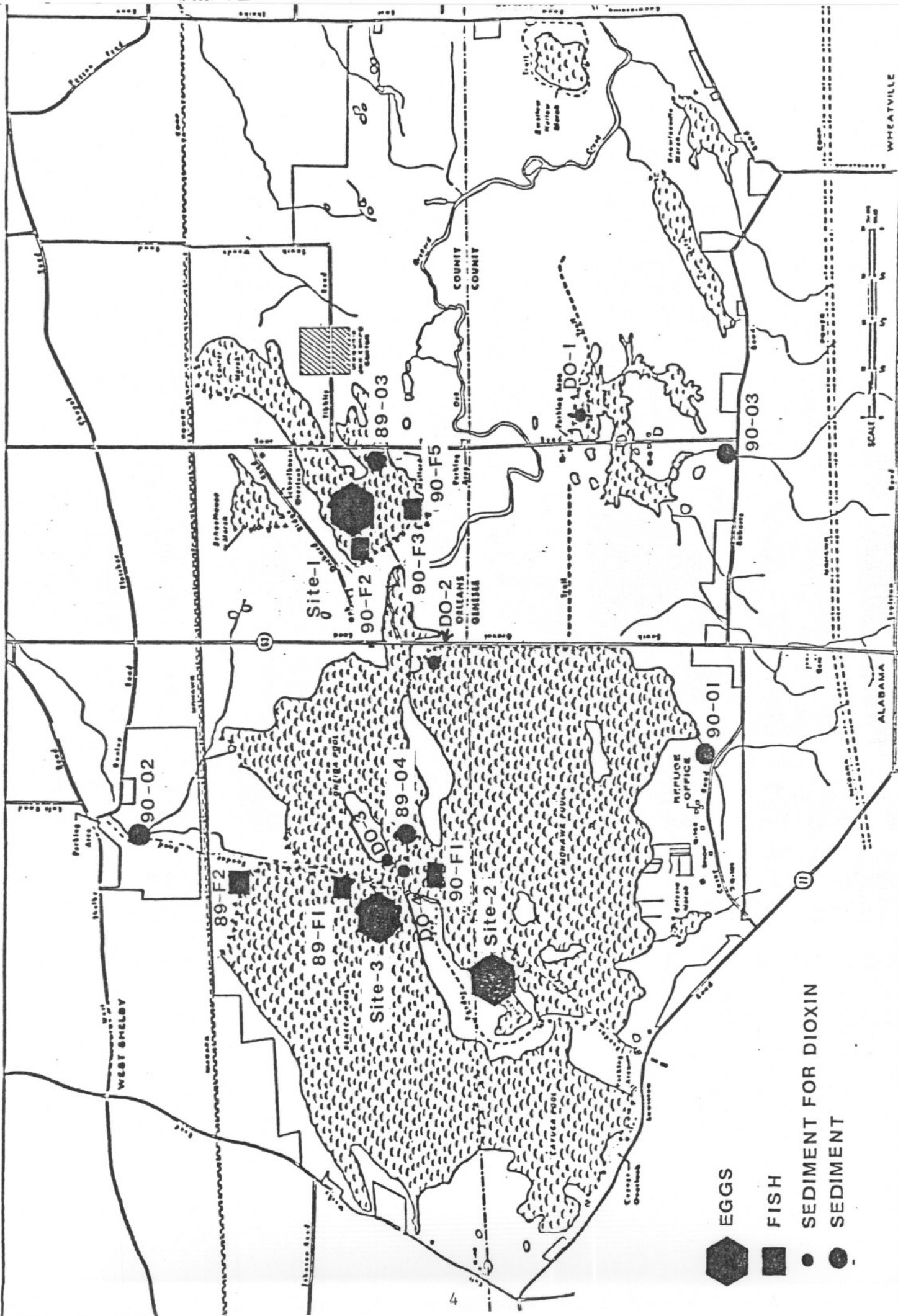


Figure 1a. Sampling Locations at Oak Orchard Wildlife Management Area.

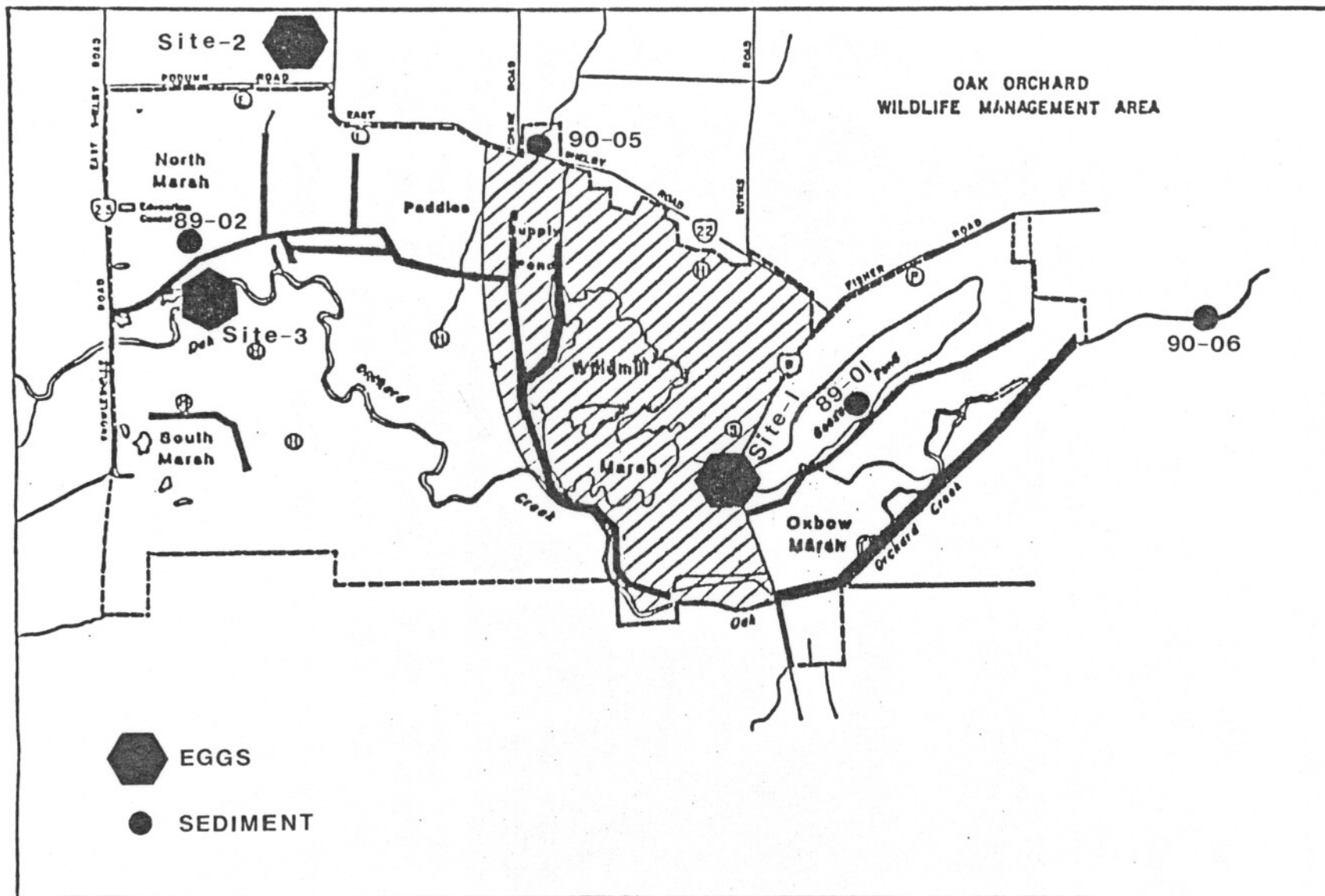
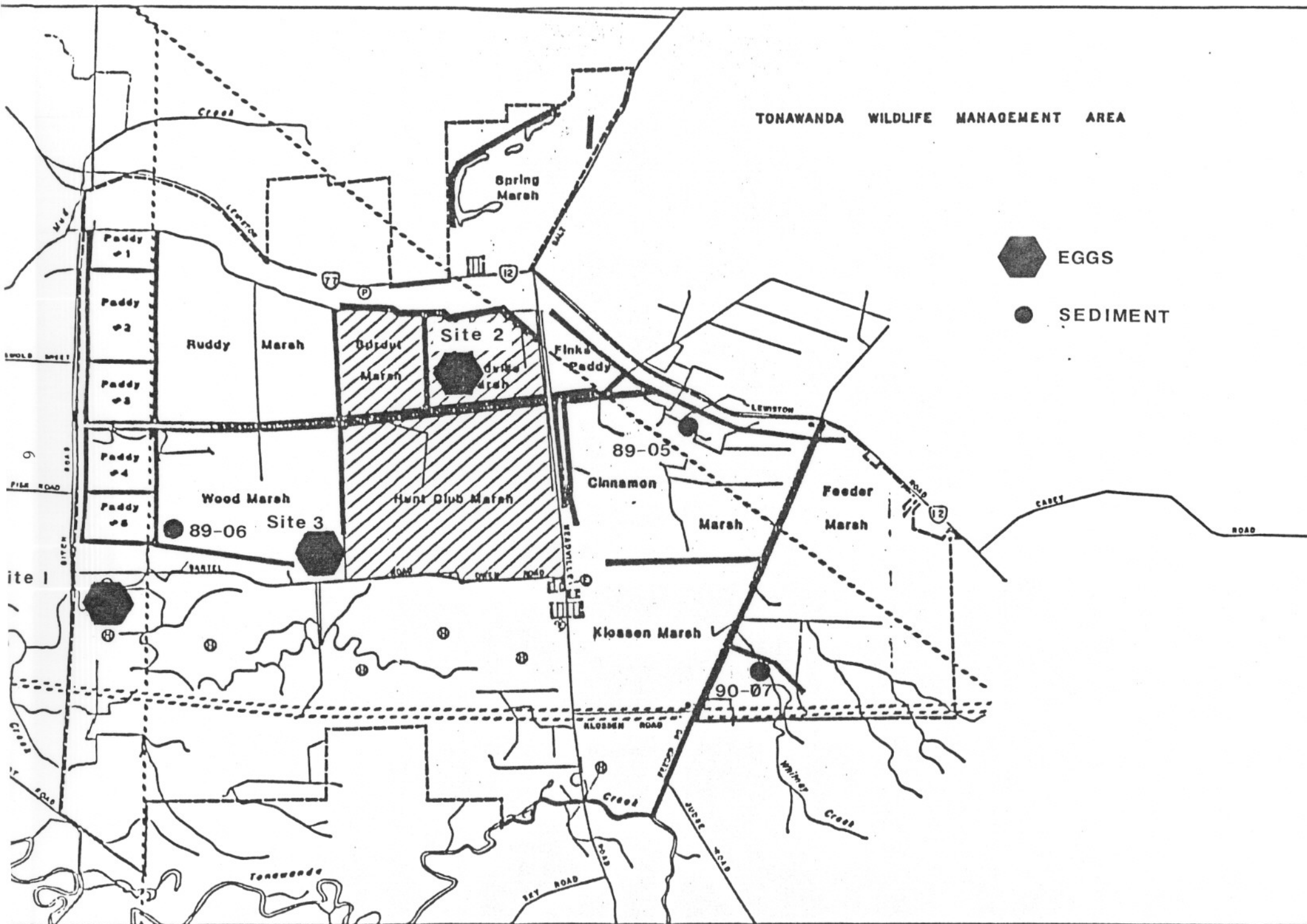


Figure 1b. Sampling Locations at Tonawanda Wildlife Management Area.

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Each egg was then cut in half by scoring around the equator with a scalpel and carefully separating the halves. The egg contents were transferred to chemically clean glass jars and frozen until analyzed. Three eggs were pooled to make one sample, thus, each pooled sample represented one site. Nine pooled samples of wood duck egg contents were submitted for analysis, three from each management area. Five pooled samples of hooded merganser egg contents were submitted for analysis, one of which represented only two eggs.

After obtaining the egg contents, the interior of each shell half was rinsed with tap water taking care not to remove the membrane. The shell halves were air dried and stored in recycled egg cartons in a dry, room temperature environment for at least 30 days. Eggshell thickness was measured with a modified dial micrometer with rounded contacts. Thickness measurements (to one thousandth of a millimeter) were taken at five points around the equator of each shell half. The reported measurement is the average of the 10 measurements per egg.

Egg harvest and sample preparation protocol are described in Appendix C.

Fish

Brown bullheads (*Ictalurus nebulosus*) and yellow perch (*Perca flavescens*) were collected by electroshocking on June 28 -29, 1990 at two sites (Mohawk Pool and Ringneck Marsh) on the Iroquois NWR. Three composite samples of five bullheads each and one composite sample of five perch were submitted for analysis. Also submitted were two composite samples of five bullheads each that were collected by electroshocking at two sites on the refuge in September of 1989 and archived at the New York Field Office to be submitted with the 1990 collections. Fish were collected, processed, stored, and shipped according to the National Contaminant Biomonitoring Program protocol.

Residue Analysis

Inorganic analysis of waterfowl eggs and turtle liver tissue was carried out by Hazleton Laboratories, Inc. in Madison, Wisconsin. Inorganic analysis of the 1989 sediments was carried out by the Environmental Trace Substances Research Center in Columbia, Missouri. Inorganic analysis of the 1990 sediments and the fish composites was carried out by the Research Triangle Institute, Research Triangle Park, North Carolina.

Organochlorine analysis of eggs, turtle fat, fish composites, and 1990 sediments was carried out by the Mississippi State Chemical Laboratory, Mississippi State, Mississippi. Organochlorine analysis of the 1989 sediment samples was carried out by the Texas A & M Research Foundation, College Station, Texas. Dioxin analysis of sediment samples was done by Wright State University, Dayton, Ohio.

Only values at or exceeding the limit of detection (LOD) are reported for all tissue matrices submitted for analysis. The data for turtle tissue and wood duck eggs was tested statistically for site differences if a given contaminant was found at, or above, the LOD in every sample. Statistical analysis was

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carried out on log transformed data using the SPSS/PC program. Arithmetic means are reported for all data.

RESULTS AND DISCUSSION

Sediment

Sediment analysis data showed that, except for one 2,3,7,8-TCDD observation, no organochlorines were found at levels at or above the detection limit of the method used, either on the refuge complex or in the tributaries. This is somewhat surprising in light of the historically agricultural nature of the surrounding watersheds and the fact that Oak Orchard Creek which flows through, and serves as the main water supply for, the entire complex originates in the heart of a muck farming area approximately 10 miles away.

The single 2,3,7,8-TCDD observation (Table 1) is an extremely low concentration. Eisler (1986) reported that sediments in Lake Winthrop, Massachusetts had 5.9 ppt 2,3,7,8-TCDD and sediments from a New York Bight "control" site and a Passaic River, New Jersey test site had as much as 79.3 ppt and 759 ppt 2,3,7,8-TCDD respectively (Pruell et al., 1990).

Table 1. Results of Dioxin Analysis of Selected Sediment Samples from Iroquois National Wildlife Refuge - 1990.

Dioxin Isomer	ppt (Dry Weight)			
	DO-1 Onondaga Pool	DO-2 Mohawk Pool	DO-3 Oneida Pool	DO-4 Mohawk Pool
2,3,7,8-TCDD ^{a/}	0.262	---	---	---

^{a/} 2,3,7,8-Tetrachlorodibenzo-p-dioxin.

Dioxins are not water soluble and do not migrate significantly in groundwater. It would be purely speculative to correlate a single, low dioxin observation with the 2,4,5-T burial site. Particularly since the Environmental Protection Agency (EPA) sampled sediment from Long Marsh directly south of the burial site in 1985 and found no 2,3,7,8-TCDD, or any other priority pollutant compound. It would seem more likely that the trace of dioxin found in the Onondaga Pool is a residue from spraying activities that were terminated more than 20 years ago.

The inorganic concentrations in the sediments show a great deal of variability from site to site (Tables 2 & 3). Levels of several of the elements overlapped thresholds established by the EPA in their Great Lakes Criteria,

Table 2. Heavy Metals/Trace Elements in Sediments from Iroquois NWR (IR), Oak Orchard Wildlife Management Area (OOWMA), and Tonawanda Wildlife Management Area (TOWMA) - 1989.

Element	ppm (Dry Weight)					
	IR		OOWMA		TOWMA	
	Ringneck Marsh 89-03	Oneida Pool 89-04	Goose Pond 89-01	North Marsh 89-02	Cinnamon Marsh 89-05	Wood Marsh 89-06
Al	4520	11100	6500	12600	7920	23100
B	--	12	--	5	9.3	6.7
Ba	26.2	84.4	66.9	120.0	39.2	125.0
Be	--	0.54	0.20	0.47	0.30	0.71
Cd	--	0.4	0.40	--	--	--
Cr	5.3	18.0	8.8	17.0	10.0	24.0
Cu	2.0	20.0	11.0	12.0	6.6	14.0
Fe	4280	17000	5810	10200	7360	17100
Mg	901	4010	2050	3900	1530	3430
Mn	75.2	124.0	91.4	99.5	84.9	217
Ni	4.0	17.0	5.3	11.0	7.0	19.0
Pb	5.0	19.0	13.0	10.0	10.0	23.0
Sr	8.2	185.0	27.9	46.8	28.4	37.9
V	6.4	20.0	10.0	17.0	13.0	29.0
Zn	19.0	95.9	55.4	90.6	40.3	76.9
As	1.0	4.1	1.4	4.1	1.8	2.8
Hg	0.03	0.04	0.04	0.053	0.04	0.054
Se	--	0.97	0.51	0.44	0.20	0.30

Table 3. Heavy Metals/Trace Elements in Tributary Sediments - 1990.

Element	ppm (Dry Weight)					
	Casey Road 90-01	Oak Orchard Creek Outlet 90-02	Roberts Road 90-03	East Shelby Road 90-05	Oak Orchard Creek 90-06	Whitney Creek 90-07
Al	24900	17800	35900	26200	13700	5700
B	88.2	26.6	77.3	39.3	51.4	68.9
Ba	234	352	287	331	150	358
Be	1.11	0.474	1.05	0.621	1.10	1.18
Cd	1.01	--	--	--	--	--
Co	19.3	9.93	19.7	12.8	11.8	14.9
Cr	42.6	15.6	37.8	23.5	38.0	40.4
Cu	23.0	7.17	31.3	12.5	7.82	21.6
Fe	25800	9420	27800	13700	25000	24000
Mg	1470	2160	7720	6510	2220	879
Mn	347	229	679	282	178	287
Ni	24.4	7.39	21.1	10.6	18	28.8
Pb	44.7	22.2	61.0	43.1	27.4	26.6
Sr	258	117	381	115	90.0	54.8
V	72.6	25.4	68.0	41.5	67.6	76.5
Zn	157	88.8	373	177	269	73.3
As	5.19	2.22	7.43	3.37	3.12	8.36
Hg	0.117	--	0.0636	0.0363	--	--
Se	0.672	0.354	0.600	--	--	--

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Sediment Pollution Categories/Bulk Sediment Analysis Ranges. However, their criteria are derived from extensive analysis of harbor sediments where inorganic inputs have been correlated with the impact of human industrial activities. As the refuge wetland complex is located in a rural setting it is difficult to make a distinction between man-caused and natural background levels of various inorganic constituents in sediments. Virtually all of the mean constituent levels in this survey were equivalent to or below the mean levels characteristic of soils and other surficial materials throughout the United States in general as measured by the U.S. Geological Survey (Shacklette et al., 1971).

Two elements that have gained much notoriety in recent years are selenium and mercury. In this survey, all selenium levels were similar to, or less than, those found in sediments from the Great Lakes and freshwater lakes in Canada, and well below those found in lake sediments in the vicinity of smelter operations (Eisler, 1985a). All mercury levels were similar to background levels found in sediments from uncontaminated areas in the north central U.S. (Eisler, 1987).

Aluminum levels appear to be excessive in several of the tributary sediments but none-the-less are still one-half or less of the average of 66,000 ppm for surface materials in the U.S. (Shacklette et al., 1971) and, it should be noted, aluminum is the third most abundant element in the earth's crust.

Snapping Turtles

Few organochlorine compounds were found in the snapping turtle fat (Table 4). Polychlorinated biphenyls (PCB's) were detected in 11 of the 15 turtles, but with the exception of a single turtle from the refuge, the levels were one-half or less of the U.S. Food and Drug Administration (FDA) action level of 5.0 ppm for edible fillets (with skin) of fish, well below levels reported in snapping turtles from eastern and northern New York a decade ago (Stone et al., 1980), and well below levels in snapping turtles from a control site in Maryland and two test sites in New Jersey (Albers et al., 1986).

DDE, a metabolite of DDT, and dieldrin were again found in all the turtles but at low levels, comparable to those found in 1988, and much lower than those found in snapping turtles by Stone and his colleagues. However, turtles from the refuge had significantly more DDE in their body fat than did turtles from the Tonawanda Wildlife Management Area. The reason for this site specific difference is not known.

Oxychlordane, alpha-chlordane, and t-nonachlor again were present at levels similar to those measured in 1988 but turtles from the refuge had significantly more alpha-chlordane and t-nonachlor in their fat than did turtles from either the Tonawanda or Oak Orchard Wildlife Management areas. As with the DDE, the reason for this is not known. However, we are dealing with marginally elevated levels overall, and it may well be that these site specific differences do not have biological significance.

Table 4. Organochlorine Compounds in Fat of Snapping Turtles from Iroquois NWR (IR), Oak Orchard Wildlife Management Area (OOWMA), and Tonawanda Wildlife Management Area (TOWMA) - 1989.

Compound ^{a/}	IR		OOWMA		TOWMA	
	Mean	Range	Mean	Range	Mean	Range
PCB (total)	4.5 ^{1/}	0.59 - 14.0	1.4 ^{1/}	0.8 - 2.7	1.3 ^{2/}	0.9 - 1.6
p,p'-DDE	0.51 A	0.23 - 0.87	0.28 A	0.13 - 0.48	0.14 B	0.07 - 0.24
Oxychlordane	0.20	0.03 - 0.72	0.07	0.03 - 0.18	0.04	0.02 - 0.05
α-chlordane	0.06 A	0.03 - 0.14	0.02 ^{2/} B	0.01 - 0.03	0.01 B	0.01 - 0.02
t-nonachlor	0.09 A	0.02 - 0.21	0.03 B	0.02 - 0.07	0.03 B	0.02 - 0.05
Dieldrin	0.08 ^{1/}	0.02 - 0.23	0.02 ^{2/}	0.02 - 0.03	0.02 ^{1/}	0.01 - 0.02
Hept. Epoxide	0.04	0.01 - 0.11	0.02	0.01 - 0.05	0.02	0.01 - 0.03

^{a/} All means calculated from 5 observations unless noted otherwise.

^{1/} Mean of 4 observations.

^{2/} Mean of 3 observations.

Means sharing the same letter on the same line are not statistically different (Student-Newman-Keuls procedure ($p \leq 0.05$)).

Notes: Hexachlorobenzene was found at or only slightly above the detection limit in 9 of 15 turtles.

Mirex (range 0.01 - 0.13 ppm (wet weight)) was found in 5 turtles.

p,p'DDD (range 0.01 - 0.12 ppm (wet weight)) was found in 6 turtles.

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Mirex, which was not detected in 1988 was present in the fat of 5 of the 15 turtles but four of the values were nearly an order of magnitude less than the FDA action level of 0.1 ppm while the remaining value (0.13 ppm) was just over the action level.

Table 5 presents the results of inorganic analysis of snapping turtle livers. The profile is similar to that of 1988 although aluminum levels appear to be considerably higher. Aluminum was measured in only 7 of the 15 turtles and the single extremely high value (92 ppm at the refuge) was more than three times higher than the next highest value. Although all values were much higher than those reported for livers of smallmouth bass (*Micropterus dolomieu*) from a southern reservoir (Brumbaugh and Kane, 1985) the values were similar to those of snapping turtles at the Montezuma National Wildlife Refuge in Central New York.

Liver mercury and selenium levels were very similar to 1988 levels and were below or within the range of background values for a variety of species (Eisler, 1985a, 1987). The apparent, but not statistically significant, elevation of the mean mercury level in livers of turtle from the Tonawanda Wildlife Management Area is due to a single high value of 10.6 ppm (2.97 ppm fresh weight (FW)). No other turtle from anywhere on the wetland complex had a level exceeding 3.87 ppm dry weight (1.1 ppm FW) and the majority were well below 1.0 ppm FW.

Boron, strontium and tin were not determined in 1988 but were detected in the 1989 samples. Boron was found in 7 of the 15 livers while strontium was found in all 15. Eisler (1990) presents limited data on boron concentrations in various tissues of freshwater organisms which shows that the turtle liver concentrations are lower than those found in whole mosquitofish and aquatic insects from both Kesterson National Wildlife Refuge, California, which has been contaminated by selenium-laden irrigation drainwater, and the Volta Wildlife Area, California, the control area for the Kesterson studies.

The significance of the strontium levels is not known, as there is little data for comparison, but they may be related to the background levels in the sediments. It is known, however, that strontium is non-toxic to organisms except at very high concentrations. The exception would be the radioisotope strontium-90 which is a strong beta-emitter and concentrates in bone tissue. No radionuclide testing was carried out on sediment samples in this survey but at least a preliminary screening of sediments may be warranted in the future.

The levels of tin in snapping turtle livers were considerably higher than levels in various tissues of a number of aquatic species (Eisler, (1989)). The mean level of total tin in the turtle livers was 2.2 ppm (FW). This is up to 22 times higher than in soft tissue of a number of saltwater invertebrates, 4.4 times higher than in muscle tissue of northern pike (*Esox lucius*) from Lake Erie (0.5 ppm FW), within the range of muscle tissue values for northern pike from Manitoba, Canada (0.7-5.4 ppm FW), up to 22 times the level in livers of 82 species of saltwater fish, and 55 times the mean level in livers of farmed Atlantic salmon (*Salmo salar*). There are no comparative data for tin in snapping turtles so little can be inferred about the metabolic

Table 5. Heavy Metals/Trace Elements in Livers of Snapping Turtles from Iroquois NWR (IR), Oak Orchard Wildlife Management (OOWMA), and Tonawanda Wildlife Management Area (TOWMA) - 1989.

Element ^{a/}	IR ppm (Dry Weight)		OOWMA ppm (Dry Weight)		TOWMA ppm (Dry Weight)	
	Mean	Range	Mean	Range	Mean	Range
Al	49.6 ^{1/}	7.22 - 92.0	9.3 ^{2/}	5.7 - 12.1	21.6 ^{1/}	14.9 - 28.2
B	3.9 ^{1/}	1.9 - 6.0	6.5 ^{1/}	3.5 - 9.6	3.3 ^{2/}	3.2 - 3.5
Cd	0.35 ^{3/}	---	0.36 ^{3/}	---	0.61 ^{3/}	---
Cu	6.4	3.8 - 8.4	4.6	2.3 - 6.3	7.5	2.6 - 12.0
Fe	761	206 - 1860	791	341 - 1700	383	265 - 600
Mn	5.0	3.0 - 6.6	4.9	2.8 - 7.3	6.3	3.0 - 8.2
Mg	481	332 - 596	473	358 - 644	503	307 - 710
Sr	2.3	1.9 - 2.6	2.6	1.3 - 5.1	2.9	1.1 - 4.8
Sn	8.1	5.3 - 10.8	8.2	5.4 - 13.0	10.5	5.8 - 16.2
Zn	122.0	69.8 - 180.0	90.0	74.6 - 106.0	94.0	54.7 - 134.0
Se	1.9	0.58 - 3.2	2.0	0.97 - 4.10	1.8	1.1 - 3.6
Hg	1.12	0.226 - 3.87	0.77	0.549 - 1.210	3.29	1.05 - 10.60
% Moisture	73.4	65.4 - 78.2	72.4	66.2 - 78.4	71.9	62.5 - 81.0

^{a/} All means calculated from 5 observations unless noted otherwise.

^{1/} Mean of 2 observations.

^{2/} Mean of 3 observations.

^{3/} One value only.

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significance of the levels found in this study. Particularly since turtles are basically static reservoirs for accumulating various compounds at levels that may or may not be accurate reflections of their significance to other, more mobile, organisms.

Table 6 presents a comparison of the levels of 4 trace elements in the snapping turtle livers from this study with levels found in the livers of snapping turtles from a Maryland control site and two New Jersey test sites (Albers et al., 1986). Cadmium concentrations were similar across the board and well below the 10 ppm (FW) threshold for vertebrate liver suggested by Eisler (1985b). Copper concentrations were slightly higher than the Maryland controls but well below the levels in turtles from the New Jersey test site. Mercury levels were generally lower except when TOWMA females were compared to females from the Maryland control area and males from the freshwater site in New Jersey. Zinc levels were similar but tended to be lower in the turtles from the refuge complex.

There were no statistically significant differences attributable to the area of collection for any of the inorganic elements detected in turtle livers in this study.

Waterfowl Eggs

Table 7 summarizes the results of organochlorine analysis of wood duck egg and hooded merganser egg contents. The only compound found in all the wood duck and hooded merganser egg samples was DDE. The levels in wood duck eggs are quite low, although slightly higher than for wood duck eggs from the Erie National Wildlife Refuge (Erie NWR), Crawford County, Pennsylvania (Rice, 1990). Except for traces of dieldrin in two samples from the Tonawanda Wildlife Management Area and traces of DDT in two samples from the Oak Orchard Wildlife Management Area, no other organochlorines were detected at measurable levels in the wood duck eggs.

Levels of DDE in hooded merganser eggs ranged from 10 to 35 times higher than in the wood duck eggs but when compared to historical data were similar to or lower than in merganser eggs from three regions of the U.S. in 1973-75 (White and Cromartie, 1977). The mean value for this survey was 0.87 ppm (FW) which is slightly higher than for mergansers from the Midwest and South-Central United States but one-half of the level those authors reported for merganser eggs from the Northeast. In addition, when compared to the DDE levels found in two eggs from the Iroquois NWR in 1975 the current level is only one-fourth as high.

All of the PCB, dieldrin, heptachlor epoxide, mirex, and chlordane isomer levels measured in merganser eggs in this survey were well below those found by White and Cromartie (1977) whether on a regional basis or just from the Iroquois NWR.

Table 7a is a comparison of thickness measurements of hooded merganser eggs from various regions of the United States over several time periods. It is interesting to note that the mean shell thickness reported here (0.628

Table 6. A comparison of the Metal Content of Snapping Turtle Livers Collected at the Refuge Complex in 1989 with Livers Collected at Two Sites in the Hackensack Meadowlands of New Jersey (NJ) and an Undisturbed Site in Maryland (MD) (Albers et al., 1986). (F = Female), (M = Male), (BW = Brackish Water), (FW = Fresh Water)

Metal	Group and (Sample Size)							
	ppm (Dry Weight) ^{1/}							
	IR(F) (15)	OOWMA(F) (15)	TOWMA(F) (15)	Md(M) (7)	Md(F) (6)	NJ(BW,M) (8)	NJ(BW,F) (3)	NJ(FW,M) (8)
Cd ^{2/}	0.35	0.36	0.61	0.29	0.22	0.45	0.35	0.32
Cu	6.40	4.60	7.50	5.2	5.81	44.20	22.38	8.35
Hg	1.12	0.77	3.29	3.70	1.70	5.82	5.50	2.41
Zn	122.0	90.0	94.0	113.6	108.5	229.0	168.6	123.2

^{1/} Data of Albers et al., (1986) transformed to ppm dry weight using moisture values given by authors.

^{2/} Only one value per site for IR, OOWMA, and TOWMA turtles.

Table 7. Organochlorine Compounds in Eggs of Wood Ducks and Hooded Mergansers from Iroquois NWR (IR), Oak Orchard Wildlife Management Area (OOWMA), and Tonawanda Wildlife Management Area (TOWMA).

Compound	Wood Duck ^{a/}			IR ^{c/} (1975)	Hooded Merganser ^{b/}		
	IR	OOWMA	TOWMA		IR	OOWMA	TOWMA ^{2/}
	ppm (Wet Weight)				ppm (Wet Weight)		
PCB (total)	--	--	--	2.37	0.47	0.30	0.75
p,p'DDE	0.050	0.036	0.050	3.24	1.27	0.31	1.10
Dieldrin	--	--	0.010 ^{1/}	0.20	0.02	0.01 ^{2/}	0.03
Oxychlordane	--	--	--		0.025	0.02 ^{2/}	0.03
Hept. Epoxide	--	--	--		0.015	0.03 ^{2/}	0.03
Mirex	--	--	--		--	--	0.05
p,p'DDT	--	0.010 ^{1/}	--	0.56	--	0.010	--
Γ-chlordane	--	--	--		0.01 ^{2/}	--	--
α-chlordane	--	--	--		0.01 ^{2/}	--	--
t-nonachlor	--	--	--		0.01 ^{2/}	--	0.01

^{a/} Means calculated from 3 observations unless noted otherwise.

^{b/} Means calculated from 2 observations unless noted otherwise.

^{c/} White and Cromaritie (1977) Mean of 2 eggs.

^{1/} Mean of 2 observations.

^{2/} One observation only.

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Table 7a. A Comparison of Hooded Merganser Eggshell Thickness (mean \pm S.E.) Over Time.*

Region or Area	Date	Thickness (mm)	N ^{1/}	Source
Midwest ^{2/}	1880-1927	0.628 \pm 0.025	6/55	White & Cromartie (1977)
Wisconsin	Pre-1947	0.614 \pm 0.009	?/44	Faber & Hickey (1973)
Wisconsin	1970	0.599 \pm 0.017	?/11	Faber & Hickey (1973)
Midwest ^{2/}	1973-1975	0.576 \pm 0.005	28/174	White & Cromartie (1977)
Iroquois Refuge New York	1975	0.589 \pm 0.006	2/14	White & Cromartie (1977)
Minnesota	1981	0.568 \pm 0.007	21/70	Zicus et al. (1988)
Iroquois Refuge New York	1989	0.628 \pm 0.032	14/14 ^{3/}	This survey

^{1/} N = Number of clutches/number of eggs.

^{2/} Midwest = Iowa, Michigan, Minnesota, North Dakota, Wisconsin.

^{3/} All eggs taken from wood duck boxes. One egg taken from each box.

* Adapted from Zicus et al.(1988).

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millimeters (mm)) is exactly that of eggs from the pre-DDT era, and greater than either that measured by Zicus in Minnesota in 1981 or by White and Cromartie for two eggs from Iroquois NWR in 1975.

The mean length and width of 14 hooded merganser eggs was 54.4 mm and 43.7 mm respectively. These dimensions compare well with mean values given by Bent (1962) for 116 eggs in various collections made around the turn of the century (53.5 mm by 44.9 mm).

The mean shell thickness of wood duck eggs measured in 1989 at Iroquois NWR was 0.340 mm and the corresponding mean length and width measurements were 52.7 mm and 39.6 mm. There is no data for comparing shell thickness but Bent (1962) gives mean length and width values of 51.1 mm and 38.8 mm respectively for 99 wood duck eggs in various collections before the turn of the century. Again, there is little difference between eggs collected in two different eras. Table 8 presents the mean physical characteristics of all eggs collected for this study.

Table 9 presents the inorganic compositions of wood duck and hooded merganser egg contents. The two species are remarkably similar in the levels of elements in their eggs with the exception of mercury. Wood duck eggs had only traces of mercury. The merganser eggs showed from 4 to 25 times as much mercury as the wood duck eggs but the mean level in their eggs of 0.9 ppm (FW) was still low, being just at the threshold level reported to cause reproductive problems in mallards (Eisler, 1987).

The wood duck data generally compared well with egg data from Erie NWR (Rice, 1990). Selenium levels in wood duck eggs from the Iroquois complex ranged from 35 to 65 percent lower than in Erie wood duck eggs but strontium levels in the Iroquois NWR wood duck eggs were 3 to 5.5 times those of the Erie NWR eggs.

The overall mean mercury level, 0.9 ppm (FW), in the hooded merganser eggs was slightly less than reported by White and Cromartie (1977) for hooded merganser eggs from the Northeast (1.01 ppm), somewhat higher than their values for birds from the Midwest and South-Central regions (0.64 and 0.62 ppm respectively), and was twice the level measured by Zicus et al. (1988) in hooded merganser eggs from Minnesota in 1981 (0.45 ppm FW).

When compared to the mean level of 1.44 ppm (FW) in hooded merganser eggs from Iroquois NWR in 1975 (White and Cromartie, 1977) the 1989 level was considerably lower.

Fish

The fish composite samples (Table 10) contained only traces of two organochlorine compounds. DDE was found in all of the composites and DDD was found in the brown bullheads collected from the Seneca Pool in 1989 and from the Mohawk Pool in 1990. No other organochlorine compounds were detected at levels exceeding the LOD.

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Table 8. Mean Physical Characteristics of Wood Duck and Hooded Merganser Eggs Collected at the Iroquois NWR (IR), Oak Orchard Wildlife Management Area (OOWMA), and Tonawanda Wildlife Management Area (TOWMA) - 1989.

Egg Measurement	Wood Duck			Hooded Merganser		
	Site ^{1/}			Site		
	IR	OOWMA	TOWMA	IR ^{2/}	OOWMA ^{3/}	TOWMA ^{4/}
Shell Thickness (mm)	0.342	0.341	0.337	0.619	0.639	0.630
Mass (gm)	45.31	45.84	46.80	59.67	60.74	59.28
Volume (ml)	42.95	43.28	44.65	54.42	54.78	53.65
Length (mm)	53.05	52.80	52.36	54.27	54.71	54.74
Width (mm)	39.23	39.60	40.04	43.62	43.82	43.57

^{1/} Mean of 9 eggs per collection site.

^{2/} Mean of 5 eggs.

^{3/} Mean of 6 eggs.

^{4/} Mean of 3 eggs.

Table 9. Heavy Metals/Trace Elements in Eggs of Wood Ducks and Hooded Mergansers from Iroquois NWR (IR), Oak Orchard Wildlife Management Area (OOWMA), and Tonawanda Wildlife Management Area (TOWMA) - 1989.

Element	Wood Duck ^{a/}			Hooded Merganser ^{b/}		
	IR	OOWMA	TOWMA	IR	OOWMA	TOWMA ^{1/}
	ppm (Dry Weight)			ppm (Dry Weight)		
Al	--	--	20.9 ^{1/}	--	--	--
Ba	3.4	3.2	4.0	3.7	3.7	5.4
B	--	--	1.6 ^{1/}	--	--	--
Cr	0.73	0.68	0.69	0.76	0.67	0.84
Cu	2.74	3.77	4.19	3.27	2.88	3.44
Fe	110.2	109.9	110.0	102.6	106.5	141.2
Mg	418.7	392.9	437.7	342.7	367.6	411.6
Mn	2.0	2.1	1.9	1.6	1.3	1.3
Sr	12.48	9.15	7.12	11.47	8.07	12.38
Zn	58.8	55.6	62.8	55.3	54.3	62.4
As	0.73 ^{1/}	--	1.00 ^{1/}	--	--	--
Hg	0.44	0.15 ^{2/}	0.17 ^{2/}	3.77	1.88	2.63
Se	0.78	0.70	0.46	0.71	0.50	0.55
% Moisture	68.2	67.6	67.2	66.0	66.6	68.9

^{a/} All means calculated from 3 observations unless noted otherwise.

^{b/} All means calculated from 2 observations unless noted otherwise.

^{1/} One observation only.

^{2/} Mean of 2 observations.

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Table 10. Organochlorines and Heavy Metals/Trace Elements in Whole Fish from Iroquois NWR - 1990.

Element	ppm (Dry Weight) ^{a/}					
	Seneca Pool 89-F1 ^{b/}	Seneca Pool 89-F2 ^{b/}	Mohawk Pool 90-F1 ^{b/}	Ringneck Marsh 90-F2 ^{b/}	Ringneck Marsh 90-F3 ^{b/}	Ringneck Marsh 90-F5 ^{c/}
p,p'DDE	0.02	0.03	0.02	0.02	0.02	0.03
p,p'DDD	0.01	0.02	0.2	--	--	--
Al	41.2	141.0	7.8	109.0	44.2	--
B	2.16	1.59	--	--	--	--
Ba	5.22	4.90	5.09	16.5	12.7	8.5
Cd	0.198	0.195	--	--	--	--
Cr	1.30	0.985	1.04	1.66	1.21	1.62
Cu	2.84	3.03	2.76	2.93	2.75	1.83
Fe	188.0	227.0	140.0	428.0	290.0	73.4
Mg	1970	1760	1810	2340	1960	2130
Mn	34.1	21.3	19.1	52.9	37.7	71.6
Pb	--	--	2.03	2.25	1.74	1.84
Sn	--	8.27	--	--	--	--
Sr	316.0	254.0	310.0	78.3	124.0	55.4
V	--	0.600	--	1.410	0.930	0.750
Zn	113.0	97.3	100.0	96.6	92.5	122.0
% Moisture	75.7	75.7	74.0	76.8	76.0	71.6
As	--	--	--	--	0.405	--
Se	1.42	0.899	1.40	1.09	1.04	0.988
Hg	0.547	0.209	0.0604	0.364	0.326	0.841

^{a/} Organochlorines are presented on a wet weight basis.

^{b/} Brown bullhead - Composite of 5 fish.

^{c/} Yellow perch - Composite of 5 fish.

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The inorganic profile of the fish composites does not show anything of concern. The lead, selenium, and mercury levels in the composites are comparable to, or lower than, levels in various freshwater species from control areas throughout the U.S. (Eisler, 1985a, 1987, and 1988). In a direct comparison the yellow perch from this survey averaged 188 mm in length which is roughly equivalent to 2-4 year old perch from the eastern Great Lakes (Scott and Crossman, 1973), yet their whole body mercury level was nearly equal (0.24 ppm FW) to that of 2-year old perch (0.22 ppm FW) from western Lake Erie (Busch, 1983). In general, the yellow perch in this survey tended to have lower inorganic values across the board than the brown bullheads. This may reflect the different feeding habits of the respective species. Brown bullheads are omnivorous bottom feeders, consuming nearly anything on or near the bottom surface layer, while perch are carnivorous, feeding largely on invertebrates and fishes taken in open water or off the bottom. Yellow perch over 150 mm long feed primarily on fishes, decapods, and dragonfly nymphs (Scott and Crossman, 1973).

The whole fish aluminum levels in the bullheads varied greatly but the two highest levels still did not approach levels measured in white suckers (*Catostomus commersoni*), another bottom feeder, from Sixmile and Threemile Creeks at Griffiss Air Force Base near Rome, New York (Ryan, 1989). Also, as pointed out by Brumbaugh and Kane (1985), whole carcass analysis of fish for aluminum can present various difficulties including wide variation between duplicate analyses and bias from including gut contents when preparing the whole fish for analysis.

In any event, the almost total lack of measurable amounts of organic contaminants and low levels of heavy metals of concern in the whole fish suggests negligible risk to the eagles from consuming fish from the Iroquois NWR pools.

CONCLUSIONS

Contaminant levels in sediments, snapping turtle tissue, waterfowl eggs, and fish from the Iroquois NWR and State Wildlife Management areas generally ranged from low to not-detectable by the methods of analysis employed.

Initially there was concern for mercury and selenium levels in snapping turtle livers but the final results indicate that concern was unfounded. Snapping turtles are long-lived, cold-blooded, basically static, organisms with small ranges and the liver itself is a concentrator of metals. Therefore, even if the data had shown elevated levels of various contaminants in the turtle tissues this would not necessarily have translated into a hazard for other, more mobile, biota but, conversely, low levels of contaminants in turtle tissue do strongly suggest that their surroundings are relatively clean.

The wood duck egg data also indicates little, if any, problem with contaminants, particularly if it holds that the composition of the egg reflects to a significant degree the quality of the vegetable and invertebrate food sources at the Iroquois NWR and the contiguous State Wildlife Management Areas.

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The hooded merganser eggs for the most part showed lower levels of various contaminants than in the past, signaling some improvement in the quality of their environment in general and their food sources in particular. However, this does not necessarily equate directly with the quality of food items at the refuge complex since we do not know of any correlation between body fat metabolism and egg laying activity in merganser hens similar to that found for the wood duck by Drobney (1982).

The whole fish analysis indicates a clean food source available to the eagles at the combined refuge and wildlife management complex. Although they will forage beyond the refuge boundary during the nesting period it is likely that they take ample advantage of the close proximity of the management pools to supply themselves and their nestlings with food.

In summary, the results of monitoring at the Iroquois National Wildlife Refuge and the associated New York State Wildlife Management Areas shows the Oak Orchard Creek wetland complex to be clean with respect to environmental contaminants of concern. There is no apparent threat to the resident breeding bald eagle pair from food sources within the refuge. However, to monitor the future contaminant status of the refuge, the contiguous wildlife management areas, and their aquatic resources, it is recommended that whole body composites of fish from at least three (3) sites on the combined wetland complex be collected and submitted for contaminant screening at intervals of no longer than three (3) years for the foreseeable future. The collections should represent a cross-section of both bottom-feeding and carnivorous species.

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APPENDIX A

STANDARD OPERATING PROCEDURE

Sediment Sampling

General Guidance

Sediment is one of the best matrices for identifying contaminant problems in an aquatic ecosystem. Sediment samples can reveal long-term trends in the quality of the overlying water, since many of the priority pollutants partition rapidly from the water column to the sediment. In general, the priority pollutants that present a particular threat to aquatic organisms because of high rates of bioaccumulation and/or toxicity will be found in greater concentrations in sediment than surface water.

Site Selection

Perform a survey of the entire area prior to sampling and select and flag the sampling sites. The primary objective of sediment sampling is to collect sediment that is most likely to reflect maximum levels of contamination. Whenever possible, sediment should be collected in areas that meet the following guidelines:

1. Collect in depositional areas that experience minimal water movement. Avoid areas affected by wave action, rapid currents or substantial scouring.
2. Fine grained sediments such as silts, clays and mucks are preferred for collection. Avoid collecting sediment with a high percentage of sand or gravel.
3. Only unconsolidated sediments should be collected since consolidated sediments may not represent recent deposition.
4. Do not collect near roads, bridges and other man-made structures.
5. Sediments collected in reference areas should have similar physical characteristics to those collected in contaminated areas.

Equipment for Sediment Collection

1. Stainless steel bucket auger
2. Stainless steel Ekman or Ponar dredge

3. Stainless steel mixing tray or bucket and mixing spatula/scoop
4. Acid washed sample containers
5. Cleaning materials to include non-phosphate biodegradable detergent, distilled water, acetone, 10% nitric acid, scrub brushes, rinse buckets, and containers for collection of used chemicals.
6. Sturdy ice chest and ice for field storage and transport.
7. Safety equipment to include surgical gloves, chemical resistant gloves, rubber boots/waders, life jackets and eye protection.
8. Survey stakes, flags, or buoys.
9. Data collection forms and Chains of Custody forms.

Equipment Cleaning

Any equipment used to collect, mix or store sediment should be cleaned prior to use and between each sampling site according to the following protocol:

- o Scrub the auger, dredge, mixing tray, bucket and spatula with a water/detergent solution. Stream water or tap water is acceptable for this step.
- o Rinse thoroughly with distilled water.
- o Scrub and rinse with acetone.
- o Allow to air dry.
- o Rinse thoroughly with distilled water.
- o Rinse with 10% nitric acid.
- o Final double rinse with distilled water.

The last two steps may be omitted if no analysis for metals is to be performed. Cleaned equipment should be wrapped in hexane washed foil when transporting it to the initial site or between sites.

Procedures

It is important to standardize sampling procedures between sample sites. The same sampler, sampling device and sampling techniques should be used at all sites. The selection of sampler type depends on sediment characteristics and water depth. For example, the Ekman dredge may be more suitable than the bucket auger for sampling soft substrates with a predominance of fines. However, it may not be suitable even if small numbers of coarse fragments are

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present that are likely to interfere with closure of the jaws of the dredge. The sampler should strive to minimize the loss of fine sediments from the sample by selecting the appropriate sampling device and taking appropriate precautions in its use.

At each sampling location, a minimum of five grab samples should be collected and placed in the stainless steel tray or bucket. Samples for volatile organic analysis should be taken directly from the bucket before mixing to minimize volatilization of the contaminants. The remaining sediment should be thoroughly mixed and samples placed into the appropriate containers.

Sediment Sample Handling

Containers for the various analyses should be filled as follows:

<u>Type of Analysis</u>	<u>Container(s)</u>	<u>Preservation/T^o</u>	<u>Max. Holding Time</u>
Volatile Organics	2, 40 ml glass septum vials	4°C	7 days
Extractable Organics*	1, 250 ml glass bottle	4°C	10 days for extraction; 40 days to analyze
Metals	1, 250 ml glass bottle	4°C	6 months
Cyanide	1, 250 ml glass bottle	4°C	14 days
Total Organic Carbon/ Grain Size	1, liter glass bottle	NA	NA

If samples are to be frozen, fill the containers 2/3 full and lay them on their side in the freezer. Collect duplicate samples in case of freezer breakage. Refrigerated samples should be filled to the top, with minimal air space remaining in the jar. One duplicate sediment sample should be collected for every ten samples.

Refrigerated sediment samples will be shipped directly to the contract laboratory within one day of sample collection. Samples should be kept on ice or refrigerated at a temperature of about 4°C from the time of collection until laboratory analysis. They should be adequately wrapped prior to shipping to prevent breakage. A sufficient amount of ice (not blue ice) to maintain the 4°C temperature should be enclosed with the samples in a sturdy ice chest (cooler). Completed chain of custody forms listing the cooler contents should be placed in a zip-lock plastic bag inside the cooler. The cooler should be wrapped securely with strong mailing tape and shipped via

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overnight mail to the laboratory. Frozen samples should be handled in the same manner, except shipped with a sufficient supply of dry ice to last at least 24 hours.

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Equipment for Sediment Collection

1. Stainless steel bucket auger
2. Stainless steel Ekman or Ponar dredge
3. Stainless steel mixing tray or bucket and mixing spatula/scoop
4. Acid washed sample containers
5. Cleaning materials to include non-phosphate biodegradable detergent, distilled water, acetone, 10% nitric acid, scrub brushes, rinse buckets, and containers for collection of used chemicals.
6. Sturdy ice chest and ice for field storage and transport.
7. Safety equipment to include surgical gloves, chemical resistant gloves, rubber boots/waders, life jackets and eye protection.
8. Survey stakes, flags, or buoys.
9. Data collection forms and Chains of Custody forms.

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APPENDIX B

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Wood Duck Box Locations and Identification Numbers - 1989

<u>Location</u>	<u>Nest Box #</u>	<u>Species*</u>
Iroquois Refuge:		
Ringneck Marsh (Site 1)	G-8A	W
	G-15A	H
	G-17A & B	W,W,H,H
Suttons Marsh (Site 2)	S1	W
	S10A & B	W,H,H
	S2	W,H
Seneca Pool (Site 3)	6A29A & B	W,W
	6A73B	W
Oak Orchard Management Area:		
Southeast Windmill Marsh (Site 1)	111	W,H
	98	W
	97	H
	96	W
North of Podunk Road and North Marsh (Site 2)	1	W
	12	W
	29	W
Along Oak Orchard Creek, South of North Marsh Dike (Site 3)	111A	W
	113	W
	118	W
	120	H
	126	H
Tonawanda Management Area:		
East of Ditch Road and South of Bartel Road (Site 1)	222	W
	225	W
	226	W
Meadeville & Sprout Marshes (Site 2)	213	H
	214	H
	215	W
	241	W
	242	W,H
Wood Marsh (Site 3)	104	W
	105	W
	106	W

*W = Wood Duck; H = Hooded Merganser

APPENDIX C

EGG PREPARATION FOR RESIDUE ANALYSIS AND
INTERPRETATION OF ANALYTICAL RESULTS

Harvesting egg contents from shells provides critical information about embryo development, and measurements allow for interpretation of analytical results. Think of the process as being performed in three stages, 1) whole egg measurements, 2) egg harvest, and 3) eggshell thickness measurements.

The supplies needed for the procedures include:

1. WHOLE EGG MEASUREMENTS -- distilled-deionized water, volumeter, egg candler, Kimwipes, laboratory balance (to 0.05 g increments), vernier caliper (graduated to 0.01mm).
2. EGG HARVEST -- glass jars of appropriate size (chemically-cleaned and with TFE cap-liners), chemically-rinsed scalpel, lead pencil, and technical pen.
3. SHELL THICKNESS MEASUREMENT -- dial micrometer with rounded contacts (graduated to 0.01 mm).

EGG MEASUREMENT PROCEDURE:

1. If possible, eggs should be candled to determine if cracks are present in the shell. Any cracked egg should not be rinsed or immersed in water as this may contaminate the sample.
2. Store eggs in a refrigerator if they cannot be processed immediately after collection. DO NOT FREEZE whole eggs since this will crack the shell.
3. If an egg is not cracked and is dirty (soil, feces) it should be cleaned with a Kimwipe and distilled-deionized water that is at, or near the temperature of the egg.
4. Write the sample ID number on both ends of the eggshell with a dull pencil (both IDs must be legible).
5. Record any remarkable characteristics of the egg (e.g. cracked, dented, discolorations, small in size, etc.).
6. Record the MASS (g) OF THE WHOLE EGG, then measure the LENGTH (mm) and BREADTH (mm) of the egg with calipers at their greatest dimensions. (To obtain an accurate measurement of length, one must ensure that the caliper jaws are parallel to the longitudinal axis of the egg. For the breadth measurement, the jaws must be held perpendicular to the longitudinal axis of the egg).

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7. Determine and record the EGG VOLUME (cm^3), the method of choice will depend on whether the shell is intact or cracked.

- A. INTACT SHELL: For eggs with intact shells, determine the EGG VOLUME using the water displacement technique outlined below.

Place a volumeter next to and above the pan of a laboratory balance. Set a collection vessel on the balance's pan under the side arm of the volumeter. Next, place a wire loop in the volumeter. Fill the volumeter with distilled-deionized water until it flows freely from the volumeter side arm (REMEMBER, the temperature of the water should be as close to the temperature of the egg as possible). When the water stops flowing, empty the receptacle and return it to the balance pan. Tare the water receptacle. Gently raise the wire loop and place the egg on it. Gently lower the egg until it is completely submersed (lower the egg as quickly as possible without overflowing the volumeter, or breaking the egg). The weight of the displaced water equals the volume (cm^3) of the egg. Repeat this procedure three (3) times for each egg and report the average value.

- B. CRACKED SHELL: For eggs that are cracked or dented, EGG VOLUME is estimated using the LENGTH and BREADTH measurements and an equation from the published literature (e.g. Westerskov 1950, and Stickel et al. 1973).

EGG HARVEST:

1. For eggs with a strong odor (indicating advanced decomposition of the contents), it is advisable to vent the egg before attempting to open it (explosions are possible). With safety glasses in place, gently insert a chemically-clean needle into the blunt end of the egg. Use gentle but steady pressure to pierce the shell.
2. Tare a chemically-cleaned jar and loosen the lid. Rest the egg lengthwise on an appropriate surface (compatible with the analyses requested). Using a sharp scalpel, gently score the egg about it's equator. Apply gentle, steady pressure and make several rotations around the egg. Once through the shell, insert the tip of the scalpel blade to cut the membrane and separate the two halves. Cut $1/2 - 2/3$ the distance around the egg. Invert the egg while pulling apart the shell halves and pour the contents into the opened jar. If necessary use a chemically clean teflon spatula to scrape any remaining contents into the jar (BE CAREFUL not to tear the shell membrane when using spatula).
3. Record the EGG CONTENTS MASS (g).

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4. Visually inspect the egg contents. Record presence or absence of an embryo, estimated age of embryo, abnormalities, etc.
 5. Label jar with SAMPLE ID and SAMPLE MASS (place one label on the lid and the other on the jar itself), and immediately store the sample in the freezer.
 6. Rinse the interior of the shell halves with tap water being careful not to tear the membrane, or erase the sample IDs. After the shells dry, use a technical pen to remark the shells with their sample IDs. Store the shells in a cool dry place for at least 30 days, or until they have attained a constant mass. (Recycled egg cartons serve as excellent storage containers for egg shells. One tip to ensure that shells do not migrate from their respective compartments, is to place a folded sheet of paper over the shells before closing the carton.

SHELL THICKNESS MEASUREMENT:

1. Determine the EGGSHELL MASS (to nearest 0.001 g) of dried shells.
2. Measure EGGSHELL THICKNESS using a dial micrometer with rounded contacts. Take thickness measurements of each shell-half along the equator at five places. Report the average of all TEN measurements as the final thickness measurement. If the membrane has separated from the shell, take measurements without the membrane but be sure to make note of this on the data sheet.
3. Calculate the Ratcliffe Index (Ratcliffe 1967) with the following formula:

$$\text{THICKNESS INDEX} = \frac{\text{EGGSHELL MASS (mg)}}{\text{EGG LENGTH (mm) x EGG WIDTH (mm)}}$$

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PESTICIDE RESIDUES IN EGGS OF WILD BIRDS:
ADJUSTMENT FOR LOSS OF MOISTURE

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*****
*
*  CORRECTION FACTOR  =  WHOLE EGG MASS (g)  *
*                        VOLUME (mL)            *
*
*****
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EXAMPLE:

<u>egg mass (g)</u>	<u>egg volume (mL)</u>	<u>reported ppm</u> <u>contaminant</u>
18.7	20.5	10

corrected value = $\frac{18.7}{20.5} \times 10 = 9.1$

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REPORTING RESULTS OF RESIDUE ANALYSES

In the reporting of residue data, consideration must be given to the nature of the data:

1. SAMPLE SIZE
2. PERCENT OF SAMPLES IN WHICH A COMPOUND OR ELEMENT IS NOT DETECTED

In general, 3 samples per site is considered to be the absolute minimum required for meaningful interpretations. As the sample size increases, so does one's confidence in one's interpretations. However, with the high cost of analyses, it is likely that the number of samples submitted will be limited.

Interpretations of small data sets are tricky at best. One technique commonly used to reduce the complications of statistically analyzing small data sets, is to report the GEOMETRIC MEAN, and 95% CONFIDENCE INTERVAL or RANGE.

Mechanically, all three statistics are calculated as for untransformed data. The only "significant" difference is, that the original data is log transformed before calculations are made, and the antilog taken before the data is reported. For a more detailed treatment of this topic, see Sokal and Rohlf (1981).

Another method used to aid in the interpretation of data is outlier analysis for small data sets (The Q-TEST). That is, what constitutes an extraneous or questionable value, (due to errors in physical measurement, equipment related errors, etc.). If a single value seems to be too high, or too low with respect to the entire data set, then it may be deleted from the statistical analysis (see Dean and Dixon, pp. 638, 1951).

With respect to the percent of non detects reported for a data set, a compound or element should be considered important for interpretation if:

1. it is detected in 60% or more of the samples analyzed

and

2. levels reported are greater than 2 times the reported detection limit.

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